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(12) **Patent:**                                    (11) **CA 2217300**

(54) SOLVENT PROCESS FOR BITUMEN SEPARATION FROM OIL SANDS FROTH

(54) PROCEDE DE SEPARATION DU BITUME D'UNE MOUSSE DE SABLE BITUMINEUX AU MOYEN DE SOLVANT

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ABSTRACT:

A paraffinic solvent is mixed with bitumen froth containing water and solids. Sufficient solvent is added to induce inversion when the mixture is subjected to gravity or centrifugal forces. The emulsion reports to the water phase and a dry bitumen product virtually free of inorganic solids, is obtained.

CLAIMS: Show all claims

\*\*\* Note: Data on abstracts and claims is shown in the official language in which it was submitted.

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# FIELD OF THE INVENTION

This invention relates to a paraffinic solvent addition method for separating water and solids from bitumen froth.

# BACKGROUND OF THE INVENTION

The present invention has been developed in connection with a plant for extracting bitumen from the Athabasca oil sand deposit. At this operation, the oil sands are surface-mined and the contained bitumen is separated from the sand and recovered using what is known as the Clark hot water extraction process ("CHWE"). (The terms "oil" or "bitumen" are used interchangeably herein to identify the hydrocarbon content of oil sand.)

The CHWE process is well known to those in the industry and is described in the patent literature. The "front end" of the process, leading up to the production of cleaned, solvent-diluted bitumen froth, will now be generally described.

The as-mined oil sand is firstly mixed with hot water and caustic in a rotating tumbler to produce a slurry. The slurry is screened, to remove oversize rocks and the like. The screened slurry is diluted with additional hot water and the product is then temporarily retained in a thickener-like vessel, referred to as a primary separation vessel ("PSV"). In the PSV, bitumen globules contact and coat air bubbles which have been entrained in the slurry in the tumbler. The buoyant bitumen-coated bubbles rise through the slurry and form a bitumen froth. The sand in the slurry settles and is discharged from the base of the PSV, together with some water and a small amount of bitumen. This stream is referred to as "PSV underflow". "Middlings", comprising water containing non-buoyant bitumen and fines, collect in the mid-section

2

1    of the PSV.

2    The froth overflows the lip of the vessel and is recovered in a launder. This

3    froth stream is referred to as "primary" froth. It typically comprises 65 wt. % bitumen,

4    28 wt. % water and 7 wt. % particulate solids.

5    The PSV underflow is introduced into a deep cone vessel, referred to as the

6    tailings oil recovery vessel ("TORV"). Here the PSV underflow is contacted and

7    mixed with a stream of aerated middlings from the PSV. Again, bitumen and air

8    bubbles contact and unite to form buoyant globules that rise and form a froth. This

9    "secondary" froth overflows the lip of the TORV and is recovered. The secondary

10   froth typically comprises 45 wt. % bitumen, 45 wt. % water and 10 wt. % solids.

11   The middlings from the TORV are withdrawn and processed in a series of sub-

12   aerated, impeller-agitated flotation cells. Secondary froth, typically comprising 40 wt.

13   % bitumen, 50 wt. % water and 10 wt. % solids, is produced from these cells.

14   The primary and secondary froth streams are combined to yield a product froth

15   stream, typically comprising 60 wt. % bitumen, 32 wt. % water and 8 wt. % solids.

16   This stream will typically have a temperature of 80°C.

17   The water and solids in the froth are contaminants which need to be reduced

18   in concentration before the froth can be treated in a downstream refinery-type

19   upgrading facility. This cleaning operation is carried out using what is referred to as a

20   "dilution centrifuging circuit".

1    More particularly, the combined froth product is first deaerated and then

2    diluted with sufficient solvent, specifically naphtha, to provide a solvent to froth ("S/F")

3    ratio of about 0.45 (w/w). This is done to increase the density differential between the

4    bitumen on the one hand and the water and solids on the other. The diluted froth is

5    then processed in a scroll-type centrifuge, to remove coarse solids. The bitumen

6    product from the scroll machine is subsequently processed in a disc-type centrifuge,

7    to remove water and fine clay solids.

8    The "cleaned" bitumen product from the dilution centrifuging circuit typically

9    contains 3 to 5 wt. % water and about 0.6 wt. % solids.

10   The underflows from the TORV, the flotation cells and the dilution centrifuging

11   circuit are discharged as tailings into a pond system. Water is recycled from this

12   pond for use as plant process water.

13   There are two significant problems associated with producing a cleaned diluted

14   froth still containing such quantities of water and solids. Firstly, one is precluded from

15   shipping the product through a commercial pipeline that is conveying discrete

16   shipments of a variety of hydrocarbon products. Such pipelines require that any

17   product shipped must contain less than 0.5 wt. % B S and W (bottom settlings and

18   water). Because of this requirement, one must upgrade the cleaned diluted froth

19   produced by the dilution centrifuging circuit in a refinery-type upgrading circuit located

20   close to the mining site, before shipping it. Providing and operating an upgrading

21   circuit at the mine site is very expensive. Secondly, there is a build-up in the

22   concentration of chlorides in plant process water that occurs over time. This build-up

23   arises from recycling water from the tailings pond to the tumbler and re-using the

24   tailings water as part of the water used as process water. In addition, the incoming oil

4

1    sands contain salt which adds to the chloride content in the process water. Keeping

2    in mind that the cleaned diluted bitumen product from the dilution centrifuging circuit

3    contains a significant fraction of plant water, chlorides are brought by this fraction into

4    the upgrading circuit. These chlorides are harmful in the upgrading circuit, as they

5    cause corrosion and catalyst fouling.

6         The industry has long understood that it would be very desirable to produce a

7    dry diluted bitumen froth product containing less than about 0.5 wt. % water plus

8    solids. Stated alternatively, it would be desirable to separate substantially all of the

9    water and solids from the froth.

10        Many potential solutions have been explored. These have included

11   electrostatic desalting, water-washing, chemicals addition, third stage centrifuging and

12   high temperature froth treatment. However, no effective and practical technique has

13   yet emerged which would produce dry bitumen with little accompanying bitumen loss

14   with the water.

15        There are various reasons why no successful technique has yet been devised

16   for cleaning bitumen froth to reduce the water plus solids content below 0.5 wt. %.

17   The major reason is that the water remaining in naphtha-diluted bitumen froth is finely

18   disseminated in the bitumen as globules having a diameter of the order of 3 microns

19   or less. The mixture is an emulsion that tenaciously resists breakdown.

1    In this background, only the CHWE process has been mentioned. There are

2    other water extraction processes - such as the known OSLO process, the Bitumen

3    process, and the Kryer process - which also produce bitumen froth which can be

4    cleaned by this invention.

5    With this background in mind, it is the objective of the present invention to

6    provide a new method for cleaning bitumen froth, produced by a water extraction

7    process, which method is effective to better reduce the water plus solids content,

8    preferably to about 0.5 wt. % or less.

9

10    **SUMMARY OF THE INVENTION**

11    The present invention is directed toward the breaking of the water emulsion in

12    bitumen froth. The invention is based on the discovery that a paraffinic solvent, if

13    added to the bitumen froth in sufficient amount, causes an inversion of the emulsion.

14    That is, the emulsion, a complex mixture of water, bitumen, solvent and solids, which

15    is initially in the hydrocarbon phase, is transferred into the aqueous phase. As a

16    result of the inversion, contained water effectively separates from the diluted froth

17    under the influence of gravity or centrifugal forces. The product is essentially dry

18    diluted bitumen, preferably having a solids and water content less than 0.5 wt. %.

19    (This product is hereafter referred to as dry bitumen.) This product is better suited for

20    upgrading as it is reduced in chloride content, relative to the product of the prior art. It

21    also meets pipeline requirements as to water plus solids content.

22    It is believed that the water globules agglomerate in the presence of the critical

23    concentration of the paraffinic solvent and acquire the capacity to segregate from the

24    hydrocarbon.

1      In a preferred embodiment, the invention involves a method for cleaning

2    bitumen froth containing water and particulate solids contaminants, said froth having

3    been produced by a water extraction process practised on oil sands, comprising:

4    adding paraffinic solvent to the froth in sufficient amount to produce a solvent to froth

5    ratio ("S/F") of at least 0.6 (w/w); mixing sufficiently to disperse the solvent in the

6    bitumen; and subjecting the mixture to gravity or centrifugal separation for sufficient

7    time to reduce its water plus solids content to less than about 0.5 wt %.  Most

8    preferably the solvent used is a mixture of low molecular weight alkanes with chain

9    lengths from about $C_5$-$C_{16}$, such as natural gas condensate, added in sufficient

10   amount to produce a solvent to froth ratio of about 1.0 (w/w).

11      The invention is characterized by the following advantages:

12      •    substantially all of the water and solids can be removed from the froth

13           by diluting it with sufficient paraffinic solvent;

14      •    bitumen losses with the separated water are only slightly lower than for

15           the conventional process;

16      •    unless the amount of solvent added is high enough to cause

17           asphaltenes to precipitate, the asphaltene content in bitumen lost with

18           the water is no higher than that normally associated with bitumen - thus

19           the lost bitumen can be recovered from the water using conventional

20           techniques; and

21      •    the new method has been shown to be effective at relatively low

22           temperatures (40 - 50°C), which raises the possibility that the extraction

23           process can be run at lower temperatures.

1    The method of this invention involves the mixing of the solvent with the

2    bituminous froth in a vessel for a sufficient time to ensure the complete dispersion of

3    the solvent into the froth. Normally, this can be carried out in a stirred tank with a

4    nominal retention time of 5 minutes. The separation itself can be carried out in the

5    same vessel by stopping the agitation and permitting the water droplets to separate

6    under the influence of gravity. In a continuous process, the separation can be

7    conducted in a separate settling vessel which is connected by piping to the mixing

8    vessel.

9    Broadly stated, the invention is directed to a method for cleaning bitumen froth

10   containing water and particulate solids contaminants, said froth having been

11   produced by a water extraction process practised on oil sand, comprising:

12   adding a sufficient amount of paraffinic solvent to the froth to induce inversion;

13   mixing the froth and the solvent for a sufficient time to disperse the solvent in

14   the froth;

15   subjecting the mixture to gravity or centrifugal separation for a sufficient period

16   to separate substantially all of the water and solids from the bitumen to produce dry

17   diluted bitumen; and

18   pumping the dry diluted bitumen through a pipeline to an upgrading circuit.

19   In another aspect the invention is directed to a method for delivering oil sand-

20   derived bitumen through a pipeline, comprising:

21   a) subjecting the oil sand to a water extraction process to obtain a bitumen

22   froth, the bitumen froth containing water and particulate solids contaminants;

23   b) cleaning the bitumen froth to remove water and particulate solids

24   contaminants, the cleaning process comprising:

1    adding a sufficient amount of paraffinic solvent to the froth to induce inversion;

2    mixing the froth and solvent for a sufficient time to disperse the solvent in the

3    froth;

4    subjecting the mixture to gravity or centrifugal separation for a sufficient period

5    to separate substantially all of the water and solids from the bitumen to produce dry

6    diluted bitumen; and

7    c) pumping the dry diluted bitumen through a pipeline to an upgrading circuit.

8

9    **DESCRIPTION OF THE DRAWINGS**

10    Figure 1 is a plot showing the residual water content remaining in the oil phase

11    over time in a gravity settling test where the bitumen froth has been diluted with

12    various solvents at conditions which are conventional: 80°C, S/F ratio 0.45 w/w. The

13    Plant 7 naphtha represents the conventional solvent used in the commercial plant

14    owned by the present assignees;

15    Figure 2 is a plot similar to Figure 1, showing the residual water content

16    remaining in the oil phase over time in a gravity settling test for runs conducted at the

17    same conditions as those of Figure 1, except that the S/F ratio was increased to 0.91

18    - of significance is the elimination of water from the oil phase at this S/F ratio when

19    heptane is the solvent used;

20    Figure 2a is a plot showing the residual water content remaining in the

21    hydrocarbon phase after treatment of bitumen froth with paraffinic solvents of different

22    molecular weights, ranging from butane to heptane at different solvent/froth ratios;

1    Figure 2b is a plot comparing the impact of three types of impurities (olefins,

2    naphthenes and aromatics) on the ability of a pure paraffin (Heptane) to produce dry

3    bitumen;

4    Figure 3 is a plot showing the residual water content remaining in the oil phase

5    after 30 minutes of settling time for runs using heptane as the solvent at different S/F

6    ratios.  Conditions:  centrifuging at 2000 rpm for 10 mins., 80°C - the results indicate

7    that inversion occurred at a S/F ratio of about 0.75 - 0.80;

8    Figure 4 is a plot showing the residual water content remaining in the oil phase

9    over time in a gravity settling test using:  (a) natural gas condensate ("NGC") as the

10   solvent for runs at different S/F ratios, and (b) the results of a single run using Plant 7

11   naphtha as the solvent at a high S/F ratio - of significance is the inversion for NGC at

12   an S/F ratio of about 1.00 to 1.20.

13

14   **DESCRIPTION OF THE PREFERRED EMBODIMENT**

15   A comparative testing program was undertaken under laboratory conditions.

16   Different solvents were added to bitumen froth as diluents.  The solvents varied in

17   aromatic and paraffin contents.  Various solvent/froth ratios were tried for each

18   diluent.  Various temperatures were tried.  After adding the solvent, the diluted froth

19   was centrifuged or gravity settled and the residual water, chloride and solids contents

20   in the bitumen fraction were determined.  The resulting data were then assessed.

21   In the course of the testing, certain discoveries were made, as described

22   below.  The inventive process is based on these discoveries.

9

1    More particularly, the test program involved the following materials and

2    procedures:

3    A single froth was used for all of the test runs.  This froth assayed as follows:

4         oil (or bitumen)    -    66.22 wt. %

5         water               -    24.59 wt. %

6         solids              -    9.65 wt. %

7    The solvents used in the test are set forth in Table 1.

Table 1

Solvents Used For Water Removal Studies From Froth

| | Class.** | Solvent | Source | Aromatics Content (%) | b.p. (°C) | Density (g/ml) |
|---|---|---|---|---|---|---|
| 1 | | | | | | |
| 2 | | | | | | |
| 3 | | | | | | |
| 4 | A | Pt. 7 Naphtha | SCL Pt. 7 | ~15% | 82 ~ 171 | 0.770 |
| 5 | A | Aromatic naphtha | Esso | ~98% | 143 ~ 186 | 0.872 |
| 6 | A | Toluene | Fisher Sci. | 100% | 111 | 0.866 |
| 7 | A | Xylene | Fisher Sci. | 100% | 139 | 0.868 |
| 8 | P | Hexane | Fisher Sci. | 0% | 69 | 0.664 |
| 9 | P | Heptane | Fisher Sci. | 0% | 98 | 0.684 |
| 10 | P | i-Octane | Fisher Sci. | 0% | 100 | 0.688 |
| 11 | P | Hexadecane | Fisher Sci. | 0% | 287 | 0.773 |
| 12 | P | Bayol 35(Light paraff.oil)* | Fisher Sci. | very low | light | 0.780 |
| 13 | | Cyclohexane | Fisher Sci. | 0% | 81 | 0.779 |
| 14 | | Cyclohexane | Fisher Sci. | 0% | 83 | 0.810 |
| 15 | * | indicates a trade mark | | | | |
| 16 | ** | A - designates an "aromatic" or non-paraffinic solvent | | | | |
| 17 | | P - designates a "paraffinic" solvent | | | | |

11

1        The solvent used in applicants' commercial operation is referred to as Plant 7

2   naphtha. This solvent is applied in the plant with a solvent/froth ratio of about 0.45. It

3   will be noted that Plant 7 naphtha has an aromatics content of approximately 15%.

4        Water contents in solvent-diluted bitumen and settled water samples were

5   determined by Karl-Fischer titration.

6        The procedure for the gravity settling runs was as follows, unless otherwise

7   described. Bitumen froth and diluent samples were separately placed into a water

8   bath operated at the temperature desired for the run. Once at temperature, samples

9   of froth and diluent were weighed out, to yield the desired solvent/froth ratio for the

10   run, and combined in a 32 ounce mixing jar. The diluent and froth in the jar were

11   mixed at 500 rpm for 10 minutes using a blade mixer.

12        Upon completion of mixing, the mixture was allowed to stand in the jar in the

13   bath to effect gravity settling.

14        Samples were taken at 0, 5, 15, 30, 60, 90 and 120 minute intervals. The

15   location of the sampling point was about the mid-point of the hydrocarbon fraction.

16   The collected samples were analyzed for water content.

17        Two samples of diluted bitumen product were collected from each run after

18   120 minutes of settling. One was analyzed for chloride content; the other was

19   analyzed for solids content.

20        The procedure for the centrifuging runs was as follows, unless otherwise

21   described. The bitumen froth and diluent samples were pre-heated to the run

22   temperature in a water bath. Once at temperature, samples of froth and diluent were

23   weighed out, to yield an 80 ml sample having the desired solvent/froth ratio, and

24   transferred into a 125 ml glass jar.

25        The glass jar was placed in a shaker and shaken rigorously for 5 minutes, to

26   mix the components.

1    The mixture was then introduced into a 100 ml centrifuge tube and spun at

2    2000 rpm for 10 minutes.

3    After centrifuging, two diluted bitumen product samples were taken.   One

4    sample was analyzed for water content.  The other was analyzed for chloride content.

5    In the Examples below, solids content of the product bitumen was analyzed by

6    the Dean Stark method.  This involves placing the samples in a porous thimble in a

7    distillation apparatus with toluene.  The sample is washed by refluxing hot toluene to

8    dissolve all of the "bitumen" which accumulates in the still pot.  Water condenses

9    overhead and is trapped in a "measuring boot" in the condenser.  "Solids" remain

10   behind in the porous thimble, and are weighed after the distillation is complete.

11   Samples of product bitumen from the Paraffin Froth Treatment process which

12   have been subjected to the Dean Stark analysis generally contained in the range of

13   0.00% - 0.15% (w/w) solids.   Conventional coker feed bitumen contains

14   approximately 1% (w/w) solids.

1  Example I

2      In this test, a group of solvents were tested at a S/F ratio of 0.45 (w/w), to

3  assess their capability to remove froth water with gravity settling. The test was run at

4  80°C. The solvents are described in Table I and identified in Figure 1.

5      As previously stated, the S/F ratio of 0.45 is that used in the commercial plant

6  dilution centrifuging circuit. Plant 7 naphtha is the solvent used in the circuit. The test

7  Temperature (80°C) is the same as that used in the plant circuit.

8      The results are tabulated in Table 2 and presented in Figure 1.

9      As shown, the solvents with high aromaticity gave equivalent or better water

10  removal when compared to the paraffinic solvent-heptane, at this S/F ratio.

11      In all of the runs, the residual water content in the diluted bitumen product after

12  120 minutes of settling was still in excess of 3%.

13      In summary, at the conventional S/F ratio, the aromatic solvents were as good

14  at inducing water separation as the paraffinic solvent; none of the solvents reduced

15  the water content below 3%.

TABLE 2

Residual Water in Hydrocarbon Phase by Gravity Settling at 80°C

Using Different Solvents at S/F Ratio = 0.45

| Settling time mins | Heptane | Pt.7 naphtha | Tol/Hep = 1/1 | Toluene | Xylene |
|---|---|---|---|---|---|
| | Water Content in Oil Phase (%) | | | | |
| 0 | 15.67 | 14.81 | 14.67 | 14.36 | 13.50 |
| 5 | 5.93 | 5.84 | 5.24 | 4.95 | 4.69 |
| 15 | 5.35 | 5.25 | 5.14 | 4.05 | 4.51 |
| 30 | 5.16 | 4.93 | 4.82 | 4.05 | 4.23 |
| 60 | 4.48 | 4.36 | 4.28 | 4.08 | 4.00 |
| 90 | 4.33 | 4.22 | 4.15 | 4.07 | 3.82 |
| 120 | 4.25 | 4.11 | 4.10 | 3.95 | 3.69 |

15

1    Example II

2        This example reports on a group of runs involving gravity settling and which

3    were carried out at 80°C using various solvents at a relatively high S/F ratio of 0.91

4    (w/w).

5        The results are shown in Table 3 and Figure 2.

## TABLE 3

Residual Water in Hydrocarbon Phase by Gravity Settling at 80°C

Using Different Solvents at S/F Ratio = 0.91

| Settling time mins | Pt.7 naphtha | Toluene | Aromatic Naphtha | Heptane |
|---|---|---|---|---|
| | | | Water Content in Oil Phase (%) | |
| 0 | 10.89 | 9.13 | 9.41 | 9.32 |
| 5 | 3.74 | 3.47 | 2.41 | 4.28 |
| 15 | 3.44 | 3.21 | 2.26 | 3.78 |
| 30 | 3.02 | 3.05 | 2.14 | <0.10 |
| 60 | 2.76 | 2.74 | 2.09 | <0.10 |
| 90 | 2.47 | 2.47 | 1.91 | <0.10 |
| 120 | 2.27 | 2.25 | 1.80 | <0.10 |

1      It will be noted that, at an S/F ratio of 0.91 (w/w), the residual water content in

2    the oil phase was reduced from about 4% (Example I) to about 2 - 2.5% for the

3    aromatic solvents tested.

4      However, the heptane run at the same S/F ratio gave a dramatically different

5    result. After about 15 minutes of settling time, an apparent inversion of the emulsified

6    water was initiated and virtually all of the emulsion settled into the water phase after

7    30 minutes of settling.

8      Heptane is a paraffinic solvent.  These runs disclose the discovery that a

9    paraffinic solvent at a sufficient S/F ratio will remove substantially all of the water from

10   diluted bitumen froth when gravity settled.

11

12   <u>Example III</u>

13      In this test, runs involving gravity settling were carried out at 80°C using

14   various solvents at increasing S/F ratios.

15      The results are presented in Table 4.

16      It will be noted that for heptane, the residual water content could be reduced to

17   a low value (0.1%) in decreasing settling time as the S/F ratio was increased above

18   about 0.80.

19      The data shows that an inversion can be obtained using heptane when the S/F

20   ratio is at least about 0.80.  This inversion is initiated in less time as the ratio is further

21   increased.

22      The Table 4 data further shows that the aromatic solvents (toluene, aromatic

23   naphtha, Plant 7 naphtha) were not capable of producing dry bitumen product at high

24   S/F ratios of 0.91 and 1.35.

18

## TABLE 4

### Residual Water in Hydrocarbon Phase by Gravity Settling at 80°C Using Different Solvents at Different Solvent To Froth Ratios

| Solvent | Heptane | Heptane | Heptane | Heptane | Heptane | Heptane |
|---|---|---|---|---|---|---|
| Solvent/Froth Ratio (w/w) | 0.70 | 0.75 | 0.80 | 0.91 | 1.35 | 1.35 |
| Water Content in Oil Phase (%) | | | | | | |
| Settling time (min) 0 | 11.88 | 11.45 | 11.36 | 9.32 | 9.29 | 8.81 |
| 5 | 4.64 | 4.44 | 4.24 | 4.28 | 4.23 | 2.28 |
| 15 | 4.13 | 1.48 | 2.96 | 3.78 | 3.82 | <0.1 |
| 30 | 3.66 | 1.04 | 0.31 | <0.1 | <0.1 | <0.1 |
| 60 | 3.36 | 0.56 | 0.11 | <0.1 | <0.1 | <0.1 |
| 90 | 3.08 | 0.26 | 0.13 | <0.1 | <0.1 | <0.1 |
| 120 | 2.71 | 0.13 | <0.1 | <0.1 | <0.1 | <0.1 |

| Solvent | Toluene | Toluene | Aromatic Naphtha | Plant 7 Naphtha | Plant 7 Naphtha |
|---|---|---|---|---|---|
| Solvent/Froth (w/w) | 0.91 | 1.35 | 0.91 | 0.91 | 1.35 |

19

TABLE 4 (Continued)

| Solvent | Toluene | Toluene | Aromatic Naphtha | Plant 7 Naphtha | Plant 7 Naphtha |
|---|---|---|---|---|---|
| | | Water Content in Oil Phase (%) | | | |
| Settling time (min) 0 | 9.13 | 8.20 | 9.41 | 10.89 | 8.03 |
| 5 | 3.47 | 2.74 | 2.41 | 3.74 | 2.71 |
| 15 | 3.21 | 2.46 | 2.26 | 3.44 | 2.40 |
| 30 | 3.05 | 2.25 | 2.14 | 3.02 | 2.08 |
| 60 | 2.74 | 2.03 | 2.09 | 2.76 | 1.71 |
| 90 | 2.47 | 1.65 | 1.91 | 2.47 | 1.47 |
| 120 | 2.25 | 1.44 | 1.8 | 2.27 | 1.22 |

20

1 <u>Example IV</u>

2      This example reports on runs involving centrifugation separation and use of

3 hexane as the solvent. The results are presented in Table 5. The runs were

4 conducted at temperatures ranging from 30°C to 60°C with increasing S/F ratios. The

5 other runs were conducted at varying temperatures with a constant S/F ratio.

6      The results indicate that inversion occurs for hexane at 60°C at a S/F ratio of

7 about 0.6. It further suggests that the S/F ratio required for inversion diminishes with

8 a lighter solvent.

9      The results further indicate that the invention is operative at temperatures

10 which are low (e.g. 40°C) relative to conventional temperatures (80°C) for dilution

11 centrifuging.

## TABLE 5

Residual Water, Chloride and Solids in Hydrocarbon Phase

After Centrifuging Using Hexane as Solvent at Different Temperatures

| Solvent | S/F (w/w) | Mixing temp. (°C) | Cent. temp. (°C) | Water (%) | Chloride (ppm) |
|---------|-----------|-------------------|------------------|-----------|----------------|
| Hexane | 0.50 | 60 | 60 | 2.95 | 24.0 |
| Hexane | 0.55 | 60 | 60 | 2.47 | 10.1 |
| Hexane | 0.60 | 60 | 60 | <0.1 | <1 |
| Hexane | 0.70 | 60 | 60 | <0.1 | <1 |
| Hexane | 0.80 | 60 | 60 | <0.1 | <1 |
| Hexane | 1.00 | 60 | 60 | <0.1 | 2.2 |
| Hexane | 0.70 | 50 | 50 | <0.1 | <1 |
| Hexane | 0.70 | 40 | 40 | <0.1 | <1 |
| Hexane | 0.70 | 30 | 30 | 0.76 | 3.8 |
| Hexane | 0.70 | 60 | 30 | <0.1 | |

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1 Example IV-A

2      This example illustrates the effectiveness of pure paraffinic solvents of

3 different molecular weights. The solvents tested were butane (C4), pentane (C5),

4 hexane (C6), and heptane (C7). Froth treatment was carried out under conditions

5 similar to those set out in Example IV. The solvent/froth ratio was varied for each

6 solvent over the range 0.3 to 0.9. The results, shown graphically in Figure 2A,

7 indicate that as the molecular weight of the solvent increased from C4 to C7, the

8 solvent/froth ratio required to produce dry (zero water concentration)_ bitumen

9 increased from slightly less than 0.5 for butane to about 0.8 for heptane. This

10 example suggests that inversion occurs at an essentially constant molar ratio of

11 solvent to bitumen. It will be appreciated from the foregoing that the molecular weight

12 of the paraffin used must be taken into consideration in setting the solvent/froth ratio.

13 At least for the paraffins tested, the trend appears to be that a higher molecular

14 weight paraffin must be used in higher mass ratios than a lower molecular weight

15 paraffin.

16 Example V

17      Table 6 illustrates the effect of temperature on water removal. Hexane was

18 used as a diluent at a hexane/froth ratio of 0.7 w/w and the hydrocarbon samples

19 were centrifuged at 2000 rpm for 10 minutes at temperatures different from the mixing

20 temperature. The data illustrate that separation of the water from the hydrocarbon

21 can be achieved at temperatures above about 30°C.

23

TABLE 6

Effect of Mixing Temperature and Centrifuging Temperature on

Separation of Water from Hexane Diluted Froth

Hexane/Froth Ratio = 0.7 w/w, Centrifuging 10 mins. at 2000 rpm

| Ratio: Mixing Temp °C/ Centrifuging Temp. °C (M°C/C°C) | M30/C30 | M60/C30 | M40/C40 | M50/C50 | M60/C60 |
|---|---|---|---|---|---|
| Water Content in Hydrocarbon, wt. % | 0.76 | <0.10 | <0.10 | <0.10 | <0.10 |

Example VI

Table 7 illustrates the solids content for the runs of Figure 2 resulting from the use of heptane solvent at 0.91 solvent/froth ratio, and residual solids contents for hydrocarbons where toluene and Plant 7 naphtha were used as diluents.

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TABLE 7

Effect of Diluent Type on Solids Removal from Froth

Settling Temperature 80°C, S/F Ratio = 0.91

</div>

| Diluent Type | Heptane | Toluene | Plant 7 Naphtha |
|---|---|---|---|
| Solids Residue in Hydrocarbon, wt. % | 0.15 | 0.75 | 0.79 |

Example VII

This example reports on runs involving centrifugation separation and use of paraffinic, cycloparaffinic and olefinic solvents at varying temperatures and a S/F ratio of 1.00 w/w.

Table 8 illustrates the effect of cycloparaffinic (cyclohexane) and olefinic (cyclohexane) solvents on water removal at solvent/froth ratios of 1.0 w/w. It is clearly shown that non-paraffinic solvents do not achieve the water removal of paraffinic solvents.

## TABLE 8

Residual Water and Chloride in Bituminous Froth Diluted with Various Hydrocarbon Solvents, After Centrifuging

| Solvent | Paraffinic Content | b.p.(°C) | Density (g/ml) | S/F (w/w) | Mixing Temp (°C) | Cent. Temp. (°C) | Water (%) | Chloride (ppm) |
|---|---|---|---|---|---|---|---|---|
| Hexane | 100% | 69 | 0.664 | 1.00 | 60 | 60 | <0.1 | 2.2 |
| Heptane | 100% | 98 | 0.648 | 1.00 | 80 | 80 | <0.1 | <1 |
| i-Octane | 100% | 100 | 0.688 | 1.00 | 80 | 80 | <0.1 | <1 |
| Hexadecane | 100% | 287 | 0.773 | 1.00 | 80 | 80 | <0.1 | <1 |
| Bayol 35* | 98+% | | 0.780 | 1.00 | 80 | 80 | <0.1 | <1 |
| Cyclohexane | 0% | 81 | 0.779 | 1.00 | 80 | 80 | 2.04 | 16.5 |
| cyclohexane | 0% | 83 | 0.810 | 1.00 | 80 | 80 | 2.36 | 19.0 |

* Trade Mark
Bayol 35 is a blend of higher molecular weight paraffins ($C_{12+}$)

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1    As shown:

2    • The paraffinic solvents (hexane, heptane, i-octane, hexadecane and

3    Bayol 35) were all successful in producing dry (0.1%) diluted bitumen

4    product.  This group of paraffinic solvents included normal paraffins,

5    isoparaffins (i-octane) and paraffin blends (Bayol 35);

6    • The cycloparaffinic and olefinic solvents were not successful in

7    producing a dry diluted bitumen product;

8    • Residual chlorides in the hydrocarbon phase were less than 1 ppm

9    when paraffinic solvents were used.  Cycloparaffinic and olefinic

10   solvents yielded higher chloride contents in the hydrocarbon, which

11   were consistent with retention of salt in the residual water.

12   The term "paraffinic solvent" is used in the claims.  This term is intended to

13   cover solvents containing normal paraffins, isoparaffins and blends thereof in

14   amounts greater than 50 wt. %.  It is not intended to include olefins, naphthas or

15   cycloparaffins.

1 <u>Example VII-A</u>

2      The experiments reported in this example examined the impact of compounds

3 such as aromatics, olefins and naphthenes on the ability of paraffins to produce dry

4 bitumen.   These tests were carried out because paraffinic solvents which are

5 inexpensive enough to use on a commercial scale are not pure paraffins.   For

6 example, natural gas condensate (NGC) contains about 83% paraffins, but also

7 significant amounts of aromatics and naphthenes.   Other tests reported on herein

8 (see Table II) demonstrated that when NGC is used as the froth diluting solvent, the

9 solvent/froth ratio required to produce dry bitumen is significantly higher than for pure

10 paraffins, at approximately 1.0 (mass/mass). The inventors believed that it was

11 important to identify compounds which are inhibitors to the action of paraffins in froth

12 treatment.   Such information could be useful for designing and producing an

13 economically viable solvent from existing process materials.

14      To minimize the effect of molecular weight, a family of compounds was

15 selected that were close together in molecular weight, as follows:

16      •     heptane (pure paraffin),

17      •     methyl cyclohexane (naphthene), and

18      •     toluene (aromatic).

1    The tests were carried out on a single froth sample using the procedure

2    outlined above for centrifuging runs.  The solvent mixtures tested contained heptane

3    with varying concentrations of the three non-paraffinic test solvents, which were

4    added on a volume/volume basis.  For each solvent mixture, a series of tests was

5    run, increasing the solvent/froth ratio until either a dry bitumen was produced, or the

6    solvent/froth ratio became too high for more solvent to be added to the test vessels.

7    The results of the tests are summarized in Tables 8a, 8b, 8c and 8d and

8    Figure 2b.  Table 8a shows that for pure heptane, dry bitumen is produced at a

9    solvent/froth ratio of 0.80.  The effect of addition of an olefin (hexane) even at 30%

10   (v/v), was relatively small.  It increased the solvent/froth requirement to produce dry

11   bitumen was from 0.8 to 1.0 (Table 8b).  A more pronounced effect was observed with

12   the addition of naphthene, methyl cyclohexane.  Added at only 10% (v/v), it increased

13   the effective solvent froth ratio from 0.8 to 1.0 (Table 8c).  At 20% and 30%

14   naphthene, the product contained measurable water at a solvent/froth ratio of 1.0.

15   The addition of an aromatic, toluene, at 10% (v/v) increased the effective solvent/froth

16   ratio to 1.2.

17   In Figure 2b, the effect of the three non-paraffinic compounds at 30% (v/v) are

18   compared in terms of the water content remaining in the treated bitumen at varying

19   solvent/froth ratios.

29

1    To summarize, this example suggests that aromatic and naphthene impurities

2 in a paraffinic solvent will significantly increase the amount of solvent required to

3 produce dry bitumen.  Olefin impurities appear to be less important.

4                                    Table 8a

5            Summary of Results, Pure Paraffin (heptane)

| Solvent to Froth Ratio | Upper Phase Density | Solvent Density | Solvent Bitumen Ratio | Density Hydro-carbon | Water Content of HC Phase |
|---|---|---|---|---|---|
| 0.49 | 0.8453 | 0.6838 | 0.6980 | 0.8452 | 0.91 |
| 0.61 | 0.8161 | 0.6838 | 1.0083 | 0.8155 | 0.37 |
| 0.71 | 0.8035 | 0.6838 | 1.1768 | 0.8035 | 0.07 |
| 0.80 | 0.8020 | 0.6838 | 1.2007 | 0.8020 | 0.01 |

Table 8b

Summary of Results, Olefin/Paraffin Mixtures

| | Hexane Concentration in Heptane | Solvent to Froth Ratio | Upper Phase Density | Solvent Density | Solvent to Bitumen Ratio | Density of Hydrocarbon | Water Content of HC Phase |
|---|---|---|---|---|---|---|---|
| 1 | | | | | | | |
| 2 | | | | | | | |
| 3 | | | | | | | |
| 4 | | | | | | | |
| 5 | | | | | | | |
| 6 | 10 | 0.50 | 0.8406 | 0.6832 | 0.7413 | 0.8399 | 1.31 |
| 7 | 10 | 0.70 | 0.8235 | 0.6832 | 0.9094 | 0.8233 | 0.25 |
| 8 | 10 | 0.80 | 0.8024 | 0.6832 | 1.1869 | 0.8024 | 0.04 |
| 9 | 10 | 0.98 | 0.7853 | 0.6832 | 1.4987 | 0.7853 | 0.00 |
| 10 | 20 | 0.50 | 0.92887 | 0.6826 | 0.2299 | 0.9283 | 1.62 |
| 11 | 20 | 0.70 | 0.8259 | 0.6826 | 0.8760 | 0.8259 | 0.21 |
| 12 | 20 | 0.81 | 0.8115 | 0.6826 | 1.0504 | 0.8114 | 0.05 |
| 13 | 20 | 1.01 | 0.8014 | 0.6826 | 1.196 | 0.8014 | 0.00 |
| 14 | 30 | 0.50 | 0.8475 | 0.6820 | 0.6743 | 0.8470 | 1.55 |
| 15 | 30 | 0.80 | 0.8056 | 0.6820 | 1.1257 | 0.8056 | 0.16 |
| 16 | 30 | 1.00 | 0.7870 | 0.6820 | 1.4455 | 0.7870 | 0.00 |
| 17 | 100 | 0.70 | 0.8427 | 0.6780 | 0.6887 | 0.8427 | 1.34 |
| 18 | 100 | 1.00 | 0.7826 | 0.6780 | 1.4693 | 0.7826 | 0.00 |
| 19 | 100 | 1.50 | 0.7659 | 0.6780 | 1.8771 | 0.7659 | 0.00 |

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Table 8c

Summary of Results, Naphthene/Paraffin Mixtures

| | Methyl Cyclo-hexane Concentration in Heptane | Solvent to Froth Ratio | Upper Phase Density | Solvent Density | Solvent to Bitumen Ratio | Density of Hydrocarbon | Water Content of HC Phase |
|---|---|---|---|---|---|---|---|
| 1 | | | | | | | |
| 2 | | | | | | | |
| 3 | | | | | | | |
| 4 | | | | | | | |
| 5 | | | | | | | |
| 6 | | | | | | | |
| 7 | | | | | | | |
| 8 | 10 | 0.51 | 0.8472 | 0.6923 | 0.7454 | 0.8453 | 2.31 |
| 9 | 10 | 0.60 | 0.8341 | 0.6923 | 0.8699 | 0.8331 | 0.77 |
| 10 | 10 | 0.70 | 0.8209 | 0.6923 | 1.0209 | 0.8206 | 0.22 |
| 11 | 10 | 0.76 | 0.8084 | 0.6923 | 1.2028 | 0.8082 | 0.06 |
| 12 | 10 | 1.00 | 0.7944 | 0.6923 | 1.4585 | 0.7944 | 0.02 |
| 13 | 20 | 0.46 | 0.8643 | 0.7009 | 0.6712 | 0.8589 | 3.00 |
| 14 | 20 | 0.79 | 0.8123 | 0.7009 | 1.2479 | 0.8119 | 0.17 |
| 15 | 20 | 1.00 | 0.7975 | 0.7009 | 1.5395 | 0.7974 | 0.03 |
| 16 | 30 | 0.50 | 0.8594 | 0.7095 | 0.7399 | 0.8567 | 2.92 |
| 17 | 30 | 0.70 | 0.8333 | 0.7095 | 1.0342 | 0.8317 | 1.59 |
| 18 | 30 | 0.80 | 0.8234 | 0.7095 | 1.1818 | 0.8221 | 0.63 |
| 19 | 30 | 0.90 | 0.8137 | 0.7095 | 1.3458 | 0.8131 | 0.29 |
| 20 | 30 | 1.04 | 0.8013 | 0.7095 | 1.6115 | 0.8012 | 0.06 |

32

## Table 8d

### Summary of Results, Aromatic/Paraffin Mixtures

| | Toluene Concentration in Heptane | Solvent to Froth Ratio | Upper Phase Density | Solvent Density | Solvent Bitumen Ratio | Density of Hydrocarbon | Water Content of HC Phase |
|---|---|---|---|---|---|---|---|
| 7 | 10 | 0.50 | 0.8564 | 0.7021 | 0.7071 | 0.8556 | 1.67 |
| 8 | 10 | 0.80 | 0.8286 | 0.7021 | 1.0085 | 0.8284 | 0.61 |
| 9 | 10 | 0.90 | 0.8160 | 0.7021 | 1.1987 | 0.8157 | 0.15 |
| 10 | 10 | 1.20 | 0.7881 | 0.7021 | 1.8056 | 0.7881 | 0.00 |
| 11 | 15 | 0.50 | 0.8527 | 0.7112 | 0.7998 | 0.8519 | 1.86 |
| 12 | 15 | 0.82 | 0.8157 | 0.7112 | 1.3222 | 0.8156 | 0.60 |
| 13 | 15 | 0.99 | 0.8069 | 0.7112 | 1.5106 | 0.8067 | 0.16 |
| 14 | 15 | 1.21 | 0.7962 | 0.7112 | 1.7907 | 0.7960 | 0.03 |
| 15 | 30 | 0.51 | 0.8711 | 0.7387 | 0.7823 | 0.8706 | 1.76 |
| 16 | 30 | 0.80 | 0.8428 | 0.7387 | 1.1988 | 0.8421 | 0.82 |
| 17 | 30 | 1.00 | 0.8389 | 0.7387 | 1.2641 | 0.8387 | 0.38 |
| 18 | 30 | 1.21 | 0.8263 | 0.7387 | 1.5485 | 0.8263 | 0.23 |
| 19 | 30 | 1.51 | 0.8110 | 0.7387 | 2.0326 | 0.8109 | 0.07 |

1   Example VIII

2   It has long been recognized that asphaltenes will precipitate in pentane. It was

3   reported by Reichert, C., Fuhr, B. J., and Klein, L. L., in "Measurement of asphaltene

4   flocculation in bitumen solutions", J. Can. Pet. Tech. 25(5), 33, 1986, that the onset of

5   asphaltene precipitation in pentane occurs when 1.92 ml/g of pentane is added to

6   Athabasca bitumen. Considering the bitumen content (66.22%) in the tested froth

7   sample, the asphaltene precipitation threshold is equivalent to 1.27 ml/g of pentane

8   for the froth sample.

9   As previously established, the minimum solvent to froth ratios for hexane

10  diluent and heptane diluent for water elimination are about 0.60 g/g and 0.80 g/g of

11  solvent based on froth, respectively. By considering the densities of the diluents,

12  these ratios are converted to 0.90 ml/g for hexane and 1.17 ml/g for heptane diluents.

13  Since asphaltene solubility in hexane and heptane is higher than in pentane, it

14  appears that asphaltene precipitation should not be significant in hexane or heptane

15  at S/F ratios close to the inversion point.

1    To further demonstrate that inversion of the emulsion and not asphaltene

2    precipitation was taking place, a test was conducted where heptane was added to

3    bitumen in different amounts and the quantities of asphaltene precipitating from the

4    solution was observed.   The results are reported in Table 9 and clearly show that

5    asphaltenes begin to precipitate from solution at ratios in excess of approximately 1.0

6    w/w heptane to froth, which exceeds the inversion value of 0.8 w/w heptane to froth

7    as obtained from Figure 3.

8                                    TABLE 9

9              Asphaltene Precipitation Observations with Heptane Diluent

10

| 11  Heptane to bitumen ratio (w/w) | 0.68 | 1.06 | 1.21 | 1.37 | 1.50 | 1.60 | 2.04 | 5.00 |
|---|---|---|---|---|---|---|---|---|
| 12  Equivalent heptane to froth ratio (w/w) | 0.45 | 0.70 | 0.80 | 0.91 | 1.00 | 1.06 | 1.35 | 3.11 |
| 13  Asphaltene precipitation at room temp. | No | No | No | No | No | little | some | lots |
| 14  Asphaltene precipitation at 80°C | No | No | No | No | No | little | some | lots |

15           This point is significant for the following reason.  There is a hydrocarbon loss with

16    the water fraction. If this loss is asphaltenes, then there is no practical way known to

17    applicants for recovering these lost hydrocarbons.

1    In conclusion, the foregoing examples support:

2    (1)    That paraffinic solvents when used as diluents for froth treatment at

3           appropriate S/F ratios will eliminate substantially all of the water and

4           chloride from froth upon separation using centrifugation or gravity

5           settling;

6    (2)    Both normal and iso paraffinic solvents are efficient in generating dry

7           diluted bitumen products;

8    (3)    Sufficient paraffinic solvent to achieve inversion is needed to produce

9           dry bitumen product - the critical S/F ratio will vary somewhat with the

10          solvent used;

11   (4)    The process works at low and high temperatures; and

12   (5)    Asphaltene precipitation does not appear to be a problem.

13

14   Example IX

15       A typical commercial solvent, which is largely paraffinic and commonly consists

16   of $C_4$ - $C_{20}$ hydrocarbons, is natural gas condensate ("NGL"). The composition of this

17   solvent is compared with the Plant 7 naphtha in Table 10, in which the composition is

18   described by various hydrocarbon classes.

<div style="text-align:center"><u>TABLE 10</u></div>

<div style="text-align:center">Typical Hydrocarbon Class Compositions of</div>

<div style="text-align:center">Natural Gas Condensate and Plant 7 Naphtha</div>

| Component | Paraffins | Naphthenes | Aromatics |
|---|---|---|---|
| Naphtha | 43% | 40% | 17% |
| Natural Gas Condensate | 83% | 12% | 5% |

Table 11 and Figure 4 illustrate water removal at different solvent/froth ratios using natural gas condensate as a solvent. In this example, water and solids were eliminated from the hydrocarbon at solvent/froth ratios exceeding 1.0 w/w.

## TABLE 11

### Water Removal Results From Froth With

### Natural Gas Condensate As Diluent By Gravity Settling at 40°C

| Solvent | | NGC | NGC | NGC | Pt.7 Naphtha |
|---|---|---|---|---|---|
| Solvent/Froth Ratio (w/w) | | 0.80 | 1.00 | 1.20 | 1.35 |
| Temperature (°C) | | 40 | 40 | 40 | 80 |
| | | Water Content in Oil Phase (%) | | | |
| Settling time (min) | 0 | 8.83 | 8.16 | 7.58 | 8.03 |
| | 5 | 7.32 | 6.79 | 6.22 | 2.71 |
| | 15 | 6.01 | 2.8 | <0.1 | 2.4 |
| | 30 | 1.75 | <0.1 | <0.1 | 2.08 |
| | 45 | 1.72 | <0.1 | <0.1 | |
| | 60 | 1.62 | <0.1 | <0.1 | 1.71 |
| | 90 | | | | 1.47 |
| | 120 | | | | 1.22 |

As shown, runs were carried out using S/F ratios of 0.80, 1.00, and 1.20. On the run having a S/F ratio of 1.00, the water removal increased dramatically (relative to S/F ratio = 0.80 run) and dry bitumen was produced. Stated otherwise, inversion was obtained using NGC at S/F ratio of 1.00 (w/w).

By comparison, a run using Plant 7 naphtha at 80°C and S/F ratio of 1.35 was unsuccessful in producing dry bitumen.

As stated, using NGC as the diluent at S/F ratios of 1.00 or greater resulted in substantially all of the water being removed from the oil. However a brownish rag layer was produced between the oil and water layers. See Figure 4 and Table 12.

TABLE 12

Rag Layers Produced During Gravity Settling with

Natural Gas Condensate as Froth Diluent

| Settling time (min) | Rag layer/(rag layer + upper oil layer); Vol % | |
|---|---|---|
| | NGC/Froth = 1.00(w/w) | NGC/Froth = 1.20(w/w) |
| 30 | 30% | 25% |
| 60 | 23% | 17% |
| 90 | 22% | 15% |
| 120 | 18% | 13% |
| 3 days | 9% | 8% |
| Composition of rag after 120 min settling | 51.97% + 48.03% water plus solids | / |

As settling was extended, the volume of the rag layer diminished. After settling for 120 minutes, the composition of the rag layer reached about 50% oil and 50% water plus solids.

When the rag layer was separated from the other layers and centrifuged at 2000 rpm for 10 minutes, the water and hydrocarbon separated, leaving oil containing less than 0.1% water.

1    Example X

2        This example reports on a run conducted in a scaled up pilot circuit using NGC

3    as the diluent.   The run was operated at 50°C and then the temperature was

4    increased over time, reaching 127°C.   The S/F ratio was maintained at about

5    1.20(w/w).

6        The pilot unit used is outlined schematically in Figure 5.

7        The results are set forth in Table 13.

8        The pilot unit consisted of a feed system where froth and diluent were pumped

9    through a heater and into a mixing vessel which had a nominal retention time of 2 - 5

10   minutes.   Pressures in the system were held at approximately 1000 Kpa.   Product

11   from the mixer was passed under pressure into the settling vessel which had a

12   nominal 15 minutes residence time.   The oil/water interface was monitored and

13   controlled by a conductivity probe.   The products, both hydrocarbon and slurry

14   underflow, were discharged from the process through coolers and then the pressure

15   released through positive displacement pumps.

16       The run continued for a period of 7-1/4 hours with approximately one-half of

17   the operating time at 50°C and the other half at 117°C (ave).

18       The results show that dry diluted bitumen could be recovered when the

19   process was operated at both temperatures.  (See Table 13.)

## TABLE 13

Froth Treatment Pilot Test Results with Natural Gas Condensate as Froth Diluent

| | Froth Flow Kg/min | Condensate Flow kg/min | Diluent Froth Flow kg/min | Settler Product kg/min | Settler Tails kg/min |
|---|---|---|---|---|---|
| Run #1 | 0.823 | 0.881 | 1.704 | 1.10 | 0.60 |
| Rune #2 | 0.823 | 0.966 | 1.788 | 1.39 | 0.40 |

| | Temperature Deg C | Mixing Speed | Pressure Kpa | Hydrocarbon Recovery (%) | Product Quality (%HC) | Chloride Removal (%) Wt. % | Hydrocarbon Solids Content |
|---|---|---|---|---|---|---|---|
| Run #1 | 49 | 500 | 1000 | 83.8 | 99.2 | 98 | 0.06 |
| Run #2 | 117 | 500 | 1000 | 97.6 | 90.7 | 77 | 0.32 |

41

## TABLE 14

### Centrifuging Results of Underflows From Pilot Runs

| Underflow Sample | From 50°C pilot run; Natural gas condensate | From 120°C pilot run; Natural gas condensate | From 120°C pilot run; Plant 7 naphtha |
|---|---|---|---|
| Density of U/F before cent. | 0.92g/ml | 0.98g/ml | |
| Upper oil after centrifuging | 33.8% | 11.8% | 9.0% |
| Rag after centrifuging | 41.2% | 3.4% | none |
| Water after centrifuging | 14.7% | 58.9% | 71.3% |
| Bottom solids after cent. | 10.3% | 25.9% | 19.7% |
| Water % in rag from cent. | 73.8% | 50.5% | / |
| Water % in recovered oil by cent. | <0.1% | <0.1% | 0.35% |

However, it was found that, at the low operating temperature (50°C), oil losses with the water and solids underflow were relatively high. At the high operating temperature (~120°C), the oil losses with the underflow were minimal. More particularly, samples of the underflow were centrifuged in a laboratory centrifuge at 2000 rpm for 10 minutes. The centrifuge contents separated into 4 layers, specifically: a clean oil layer; a viscous rag layer; a water layer; and a solids layer. The relative proportions are stated in Table 14. Most of the solids in the hydrocarbon were also removed.

1    In conclusion, the results teach that NGC can successfully be used as the

2    diluent at low and high temperatures to yield dry diluted bitumen.  However, the low

3    temperature process produces relatively low quality underflow and the underflow has

4    a relatively high rag content.

5

6    <u>Example XI</u>

7    This example provides a detailed analysis of the product bitumen and the

8    tailings which are produced in the froth treatment process described in Example X.  In

9    order to maximize the quality enhancement:

10    •    a relatively high solvent/froth (natural gas condensate) ratio of 1.58 was

11    used; and

12    •    the run was conducted at 50°C.

13    The lower temperature was chosen on the basis of Example X which

14    demonstrated that while the bitumen recovery was higher if the run was conducted at

15    117°C, the product might be of higher quality if the temperature was 49°C.  A

16    summary of the operating conditions and process performance data for the run are

17    shown in Table 15.

18    Three samples were taken from the unit:

19    •    feed to the unit before dilution (froth);

20    •    product from the unit (diluted bitumen); and

21    •    tailings from the unit.

1    The composition and properties of the three fractions were determined using

2    methodology which is well known in the field of bitumen upgrading. Analyses were

3    carried out in accordance with the methods documented in the analytical methods

4    directory of the LAN. The bitumen fraction of interest in the three samples was

5    selected to be that fraction of hydrocarbon which boils above 343°C. The diluted

6    bitumen product was sufficiently low in water and solids and was dilute enough that

7    the fraction of the hydrocarbon with bp above 343°C could be directly recovered from

8    the sample through distillation on a true boiling point distillation apparatus. The froth

9    and tailings samples both had significant water and solids contents. Therefore,

10   before distillation, they were first extracted with hot toluene in a Dean Starke

11   apparatus to isolate the hydrocarbon portion of the sample. This was then subjected

12   to the identical distillation process as the diluted bitumen sample.

13   The data from the analyses performed on the bitumen recovered from the

14   three distillations is shown in Table 16. The results show that the process yielded a

15   bitumen product which was significantly different from that obtained from the

16   conventional froth treatment process. In particular, the product bitumen from this

17   process in comparison to the standard product had:

18   •    almost no extraneous matter;

19   •    less aromatics;

20   •    less micro-carbon residue;

21   •    less nitrogen;

22   •    less sulphur;

23   •    less pentane insoluble material;

24   •    a better carbon to hydrogen ratio; and

25   •    a lower density.

26   "Extraneous matter" comprises material which can be measured as "solids" in

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1   the Dean Stark analysis, plus any other toluene-insoluble material which can be

2   recovered by filtration. It is basically a measurement of non-bitumen material.

3        There was also a drastic reduction in the viscosity as compared to the feed

4   bitumen, from 1880 - 324 mPa.s @ 100°C.

5        In summary, this example demonstrates that it is possible to produce a highly

6   purified dry bitumen product using the paraffin froth treatment process of the

7   invention. The product bitumen has a water content of less than about 0.1%, an

8   extraneous matter and solids content of less than about 0.1%, and a reduced

9   viscosity of generally less than about 500 mPa.s @ 100°C. The inventors believe that

10  a highly purified bitumen product has not heretofore existed.

11       Although the quality of the bitumen which is described in Example XI

12  happened to have been achieved using the paraffin froth treatment process under

13  the conditions outlined in Table 15 (natural gas condensate used at a solvent/froth

14  ratio of 1.58 at 50°C), the inventors believe that bitumen of equivalent purity can be

15  produced using other paraffinic solvents as outlined herein.

16       The inventors believe that the purity of the product bitumen obtained from the

17  Paraffin Froth Treatment process will provide the following benefits in upgrading:

18       •     In the conventional Froth Treatment process, employing Naphtha as a

19             diluent, the product bitumen contains inorganic solids which end up as

20             "ash" constituents in coke, as a fouling deposit on the inside of process

21             equipment and piping, or as deposits on catalyst pellets. These

22             deposits ultimately lead to shut-down for cleaning, frequent repairs, and

23             the consumption of catalyst used in the hydrocracking process. The

24             removal of virtually all inorganic solids from the product bitumen should

25             reduce the rate of fouling and catalyst poisoning.

26       •     Product bitumen which is free of inorganic solids can be channelled to

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1    primary upgrading steps that previously were precluded.  For example,

2    processes such as *Vega Combi-Cracking (VCC), which may be

3    superior in yield and quality of products made from raw bitumen, cannot

4    accept feeds containing significant solid impurities.

5    •    The product bitumen is clean enough to meet the Bottom Sediment and

6    Water (BS&W) specifications of pipeline operators.   The product

7    bitumen would therefore only need to be reduced in viscosity to meet

8    pipeline specifications.  This might be accomplished by the addition of a

9    small quantity of light hydrocarbon diluent (such as natural gas

10   condensate) or by the application of a mild "vis-breaking" technology

11   such as heat-soaking or low severity catalytic treatments.

12   •    If the product bitumen is produced under conditions which render it low

13   in viscosity, such as in Example II, the product bitumen would be

14   pumpable with none or very little dilution.  This could eliminate the

15   necessity of coupling extraction with upgrading facilities.

16   * Trade Mark

| | Table 15 | |
| --- | --- | --- |
| | Paraffin Froth Treatment Process Performance Data | |
| | Using Natural Gas Condensates As Solvent | |
| | Parameter | Value |
| | Solvent to Froth Ratio (wt/wt) | 1.58 |
| | Solvent to Bitumen Ratio (wt/wt) | 2.5 |
| | Mixer Temperature ($^{\circ}$C) | 50 |
| | Product Quality | |
| |     Hydrocarbon (%/wt) | 99.92 |
| |     Water (%/wt) | 0.08 |
| |     Solids (%/wt) | 0.00 |
| | Bitumen Recovery (%) | 87.6 |

## Table 16

Comparison of +343°C Fractions of Feed, Product and Tailings Bitumen

| Analysis | Feed Bitumen | Product Bitumen | Tailings Bitumen | Typical Coker Feed Bitumen |
|---|---|---|---|---|
| Carbon wt% | 80 | 83 | 79 | 83 |
| Hydrogen wt% | 10.1 | 10.4 | 9.5 | 10.4 |
| C/H ratio | 7.9 | 8.0 | 8.3 | 8.0 |
| % C in aromatic environment | 31.0 | 28.3 | 32.2 | |
| Sulphur wt ppm | 52400 | 47500 | 56000 | 49700 |
| Nitrogen wt ppm | 5240 | 4770 | 6600 | 5870 |
| Basic Nitrogen wt ppm | 1520 | 1340 | 1860 | |
| extraneous matter wt% | 0.83 | 0.02 | 1.43 | 0.50 |
| micro carbon residue wt% (not corrected for "extraneous matter"wt) | 16.3 | 13.7 | 24.2 | 15.0 |
| pentane insolubles wt% | 21.4 | 12.7 | 39.2 | |
| density gm/cc @60°C | 1.030 | 1.007 | 1.071 | |
| viscosity mPa.s @100°C | 1880 | 324 | 49200 | |
| % recovered @ 524°C | 35.6 | 44.2 | 29.5 | 41.5 |